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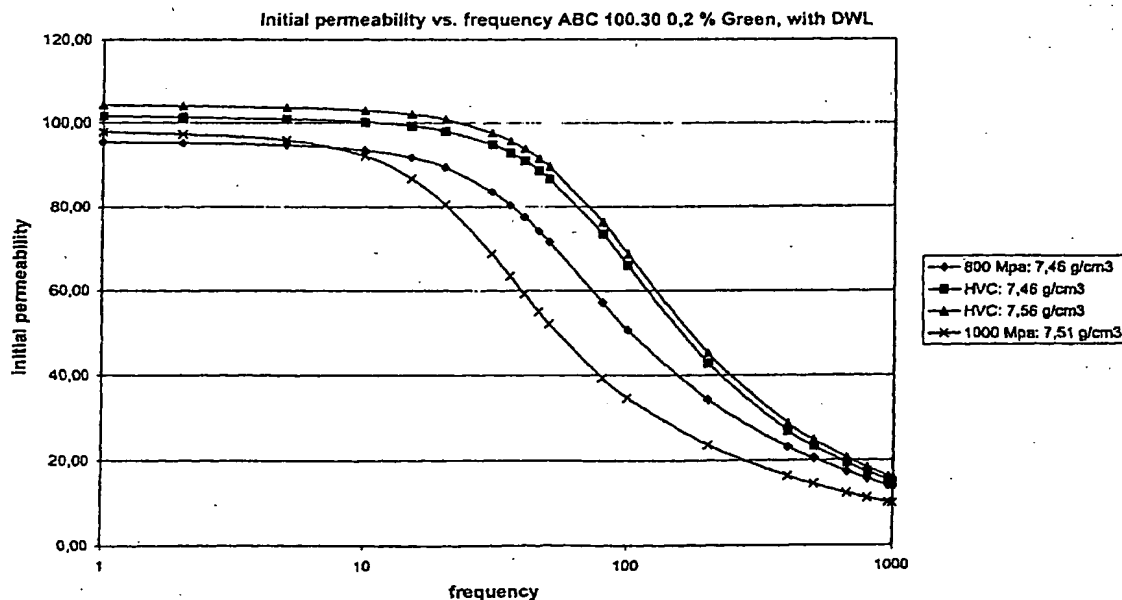
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(54) Title: METHOD OF PREPARATION OF HIGH DENSITY SOFT MAGNETIC PRODUCTS



(57) Abstract: The invention concerns a method of preparing high density compacts for soft magnetic applications comprising the steps of subjecting an iron or iron-based soft magnetic powder the particles of which are electrically insulated to compaction in an uniaxial pressure operation with a ram speed of at least 2 m/s.



MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, TR), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG)

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METHOD OF PREPARATION OF HIGH DENSITY
SOFT MAGNETIC PRODUCTS.

5

Field of the invention

This invention relates to the general field of powder
10 metallurgy. Particularly the invention is concerned with
a method of preparation of high density soft magnetic
products.

Background of the invention

15 In recent years the use of powdered metals for the manu-
facture of soft magnetic core components has expanded and
the research has been directed to the development of iron
powder compositions that enhance certain physical and
magnetic properties without detrimentally affecting other
20 properties. To this end many efforts have been made in
order to provide electrical coatings which insulate the
individual iron powder particles and many examples of
different coatings are disclosed in the art.

25 Thus according to the US patent 3 245 841 an insulated
powder is prepared by treating an iron powder with a
coating solution including phosphoric acid and chromic
acid. Insulating coatings are also described in e.g. US
5 798 177 and DE 34 39 397. According to these publica-
30 tions the coatings are obtained by treating iron based
powders with coating solutions including phosphoric acid.
The compacted product prepared from the insulated powders
is subsequently heat treated. Another type of coating is
disclosed in US 4 602 957. According to this patent a
35 magnetic powder core is prepared by treating an iron pow-
der with an aqueous solution of potassium dichromate,
drying the powder, compressing the powder to form a com-

5 pact and heat treating the compact at substantially
600°C. In other known processes soft iron particles are
coated with thermoplastic materials before pressing. The
US patents 4947065 and 5198137 teach such methods whereby
10 iron powders are coated with a thermoplastic material. A
more recent method of coating iron-based powders for soft
magnetic applications is described in PCT SE97/00283.
Thus by using different types of coatings and coating
techniques desired properties such as high permeability
15 through an extended frequency range, high pressed
strength, low core losses and suitability for compression
moulding techniques have been considerably improved
lately.

15 In addition to the development of coated powders for soft
magnetic applications efforts are also made in order to
improve the properties of non coated powders as is de-
scribed in the US patent 6 331 270.

20 It has now been found that the magnetic properties, such
as the initial permeability as a function of the fre-
quency (frequency stability), may be improved by using a
high velocity compaction (HVC) technique, which is de-
scribed more in detail below. Especially unexpected is
25 the finding that, for a given density, the initial perme-
ability at different frequencies are significantly higher
with this HVC technique and that these properties have
been observed for both insulated and not insulated powder
particles.

30

Objects of the invention

An object of the invention is to provide a method for the
preparation of high density soft magnetic products, par-
35 ticularly products having a density above 7.25, prefera-
bly above 7.30 and most preferably above 7.35 g/cm³.

A second object is to provide a compaction method adapted to industrial use for mass production of such high density products.

- 5 A third object is to provide compacted bodies having high density and high green strength.

A fourth object is to provide a soft magnetic compacts bodies having high initial permeability.

10

Summary of the invention

In brief the method of preparing such high density compacts comprises the steps of subjecting an iron or iron-based soft magnetic powder to HVC compaction with an uni-
15 axial pressure movement with a ram speed of at least 2 m/s. The particles of powder may, but must not, be electrically insulated.

Detailed description of the invention

- 20 The base powder, i.e. the non-insulated powder, may be a substantially pure water atomised iron powder or a sponge iron powder having irregularly shaped particles. In this context the term "substantially pure" means that the powder should be substantially free from inclusions and
25 that the amounts of the impurities O, C and N should be kept at a minimum. The average particle sizes are generally below 300 μm and above 10 μm . Examples of such powders are ABC 100.30, ASC 100.29, AT 40.29, ASC 200, ASC 300, NC 100.24, SC 100.26, MH 300, MH 40.28, MH 40.24
30 available from Höganäs AB, Sweden.

- An insulating coating may be applied in order to improve the properties in alternating magnetic fields. Such a coating also permits heat treatment which further en-
35 hances the magnetic properties. The coating and the coating method is believed not to be critical and the coating could e.g. be any of those disclosed above. Espe-

cially preferred are thin coatings based on phosphorus and silicone, aluminium and titanium.

5 In order to obtain the products having the desired high density according to the present invention the compacting method is important. Normally used compaction equipment does not work quite satisfactorily, as the strain on the equipment will be too great. It has now been found that
10 the high densities required may be obtained by the use of the computer controlled percussion machine disclosed in the US patent 6202757 which is hereby incorporated by reference. Particularly, the impact ram of such a percussion machine may be used for impacting the upper punch of a die including the powder in a cavity having a
15 shape corresponding to the desired shape of the final compacted component. When supplemented with a system for holding a die, e.g. a conventionally used die, and a unit for powder filling (which may also be of conventional type) this percussion machine permits an industrially
20 useful method for production of high-density compacts. An especially important advantage is that, in contrast to previously proposed methods, this arrangement driven by hydraulics permits mass production (continuous production) of such high density components.

In the US patent 6202757 it is stated that the use of the percussion machine involves "adiabatic" moulding. As it is not fully clarified if the compaction is adiabatic in a strictly scientific meaning and we have used the term high velocity compaction (HVC) for this type of compaction wherein the density of the compacted product is controlled by the impact energy transferred to the powder.

According to the present invention the ram speed should be above 2 m/s. The ram speed is a manner of providing energy to the powder through the punch of the die. No straight equivalence exists between compaction pressure

in a conventional press and the ram speed. The compaction which is obtained with this computer controlled HVC depends, in addition to the impact ram speed, i.a. on the amount of powder to be compacted, the weight of the impact body, the number of impacts or strokes, the impact length and the final geometry of the component. Furthermore, large amounts of powder require more impacts than small amounts of powder. Thus the optimal conditions for the HVC compaction i.e. the amount of kinetic energy which should be transferred to the powder, may be decided by experiments performed by the man skilled in the art. Contrary to the teaching in the US patent 6 202 757 there is, however, no need to use a specific impact sequence involving a light stroke, a high energy stroke and a medium-high energy stroke for the compaction of the powder. According to the present invention the strokes (if more than one stroke is needed) may be essential identical and provide the same energy to the powder.

Experiments with existing equipment has permitted ram speeds up to 30 m/s and, as is illustrated by the examples, high green densities are obtained with ram speeds about 10 m/s. The method according to the invention is however not restricted to these ram speeds but it is believed that ram speeds up to 100 or even up to 200 or 250 m/s may be used. Ram speeds below about 2 m/s does, however, not give the pronounced effect of densification. It is preferred that the ram speed above 3 m/s. Most preferably the ram speed is above 5 m/s.

The compaction may be performed with a lubricated die. It is also possible to include a suitable particular lubricant in the powder to be compacted. Alternatively, a combination thereof may be used. The lubricant can be selected among conventionally used lubricants such as metal soaps, waxes and thermoplastic materials, such as polyamides, polyimides, polyolefins, polyesters, polyalkoxides,

polyalcohols. Specific examples of lubricants are zinc stearate, H-wax® and Kenolube®. The amount of lubricant may vary up to 1% by weight of the powder composition.

- 5 The invention is further illustrated by the following examples:

Example 1

10 This example illustrates the possibility of obtaining high initial permeability with a soft magnetic powder (Somaloy 500 available from Höganäs, Sweden), the particles of which are electrically insulated.

15 100g powder of the powder were used in a ring tool with the dimensions Ø72/56. Both conventional compaction and HVC compaction were used. The following two mixes were tested:

Somaloy 500+ 0,2%Kenolube*

Somaloy 500+ 0%Kenolube*

20 *Lubricant available from Höganäs AB, Sweden

The compaction machine was Model HYP 35-4 from Hydropul-sor Sweden.

25 The same type of Die Wall Lubrication was used for both mixes and for both compacting methods.

The green density was determined by principle of Archimedes (1).

$$\rho = m_{air} / (m_{air} - m_w) \quad (1)$$

30 m_{air} = mass in air

m_w = mass in water

35 The height, inner and outer diameter was measured on each sample. After compaction the toroids were wound with 25 turns of insulated copper wire. The inductance of the coil was measured at 1000 and 2000 Hz with a HP 4284.A LCR- meter. The inductance was measured at low currents

(10mA) and the initial permeability was calculated from (2).

$$\mu_{in} = L \cdot l \cdot 10^{-3} / (N^2 \cdot A \cdot \mu_0)$$

L = measured inductance in μ Henry

l = magnetic length in cm

N = number of turns

10 A = cross section area in cm^2

μ_0 = permeability of free space

The samples have the same geometry and testing was made exactly the same way. At a given density an unexpected
15 difference as regards the initial permeability could be observed between HVC and conventional compacted samples as can be seen from Figure 1. The ram speeds for the HVC compaction were about 7-8 m/s.

20 Example 2

This example illustrates the possibility of obtaining high initial permeability and high frequency stability with a powder (ABC 100.30 available from Höganäs, Sweden), the particles of which are not electrically insulated before the compaction.
25

The samples have the same geometry and testing was made exactly the same way. At a given density an unexpected difference could be observed between HVC and conventional
30 compacted samples as can be seen from Figure 2 and 3. 0.2 and 0.5 % by weight, respectively, of a particular lubricant (Kenolube®) was added to the iron powder before the compaction. The stroke lengths used for the HVC compaction in Figure 2 were 85 and 100 mm corresponding to ram
35 speeds of 8 and 9 m/s, respectively. The stroke lengths used for the HVC compaction in Figure 3 were 70 and 90 mm corresponding to ram speeds of 7.5 and 8.5 m/s, respectively.

Example 3

Rings with the dimensions $\varnothing 50/30 \times 10$ mm were HVC compacted with double impacts. The ring material was Somaloy 500TM with either 0,5% or 0,1% admixed KenolubeTM. Compaction with the mix containing 0,1% Kenolube took place with the support of die wall lubrication. Table 1 gives the compaction data and the green and % of the theoretical density.

Table 1. Compaction data

Material	Impact 1 Energy/ [Nm]	Impact 2 Energy/ [Nm]	Total Compaction Energy [Nm]	Green Density [g/cm ³]	% of theor. density
Somaloy 500+0,5% Kenolube	1778	3111	4889	7.52	99.6
Somaloy 500+0,1% Kenolube	2667	4000	6667	7.68	98.9

After the HVC compaction and heat-treatment at 500°C for 30 min in air the samples were wound with 25 sense and 150 drive turns and measure in a LDJ 3500 hysteresis graph. Table 2 shows that high magnetic induction for non-sintered powder components can be achieved with HVC. A high resistivity is maintained which easily can be seen from the core loss data in table 2.

Table 2. Magnetic properties

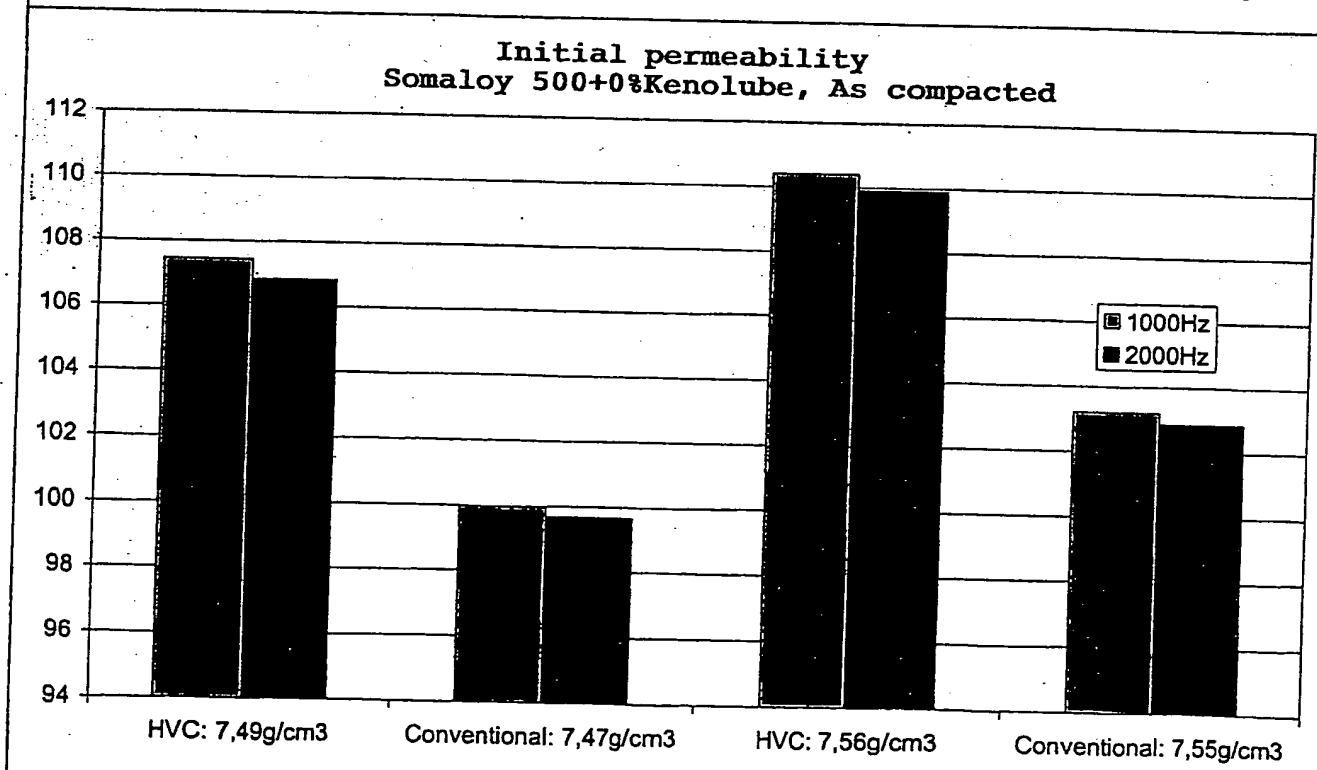
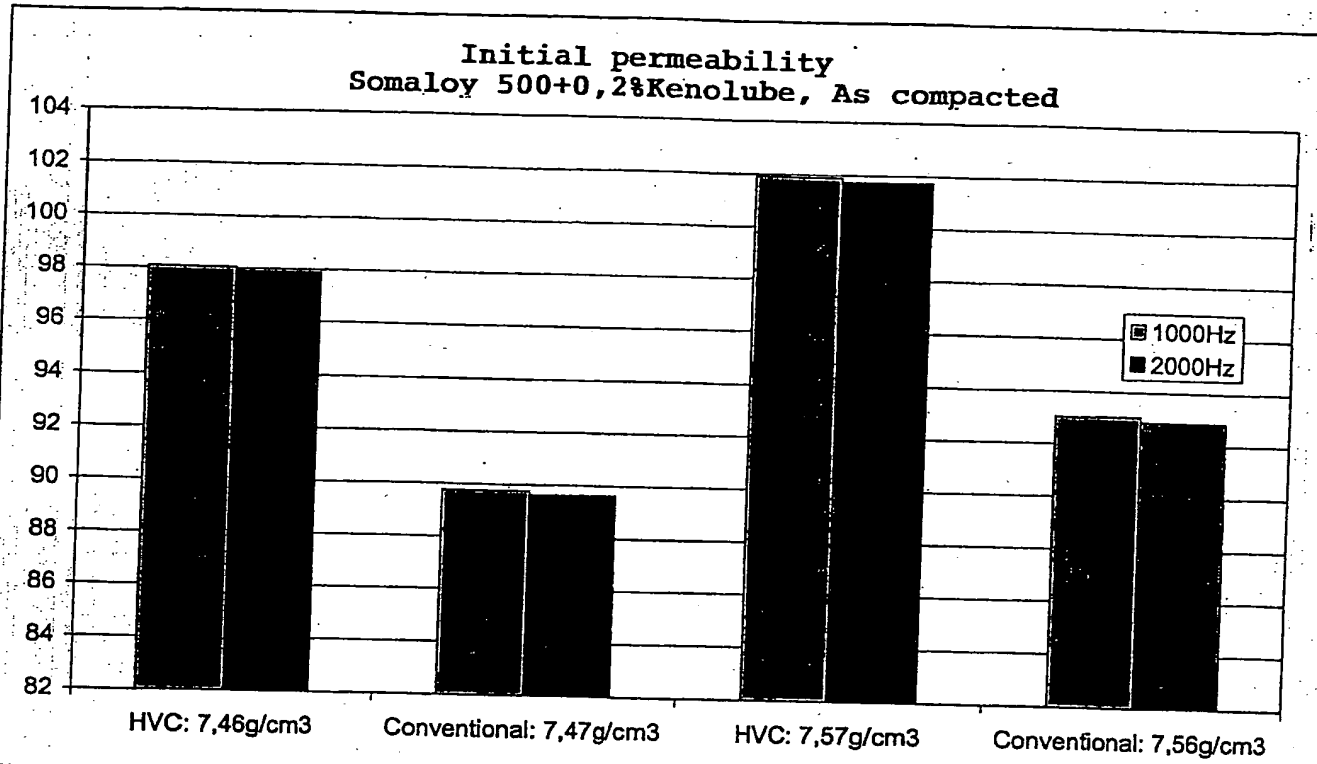
Material	B @ 10kA/m	μ_{\max}	Core loss/cycle @ IT [J/kg]	
			50Hz	200Hz
Somaloy 500+0,5% Kenolube	1,55	530	0.112	0.130
Somaloy 500+0,1% Kenolube	1,67	660	0.106	0.127

CLAIMS

1. Method of preparing high density compacts for
5 soft magnetic applications in alternating magnetic fields
comprising the steps of
subjecting an iron or iron-based soft magnetic powder to
to HVC compaction with a uniaxial pressure movement with
a ram speed of at least 2 m/s.
- 10 2. Method according to claim 1 characterised in
that the compaction is performed at a ram speed above 3,
preferably above 5 m/s.
3. Method according to any one of the claims
1-2 characterised in that the compaction is controlled by
15 the impact energy transferred to the powder.
4. Method according to any one of the preceding
claims characterised in that the compaction is performed
as warm compaction.
5. Method according to any one of the preceding
20 claims for the preparation of compacts having a density
above about 96 % of the theoretical density.
6. Method according to any one of the preceding
claims for the preparation of compacts having a density
above about 98 % of the theoretical density.
- 25 7. Method according to any one of the preceding
claims characterised in that the particles of the powder
are electrically insulated.
8. Method according to any one of the claims 1
to 8 characterised in that the compaction is performed in
30 a lubricated mould with or without internal lubricant.
9. Method according to any one of the claims
1-9 characterised in that the compaction is performed
with a powder including at most 1, preferably at most
0.5% by weight of a lubricant.

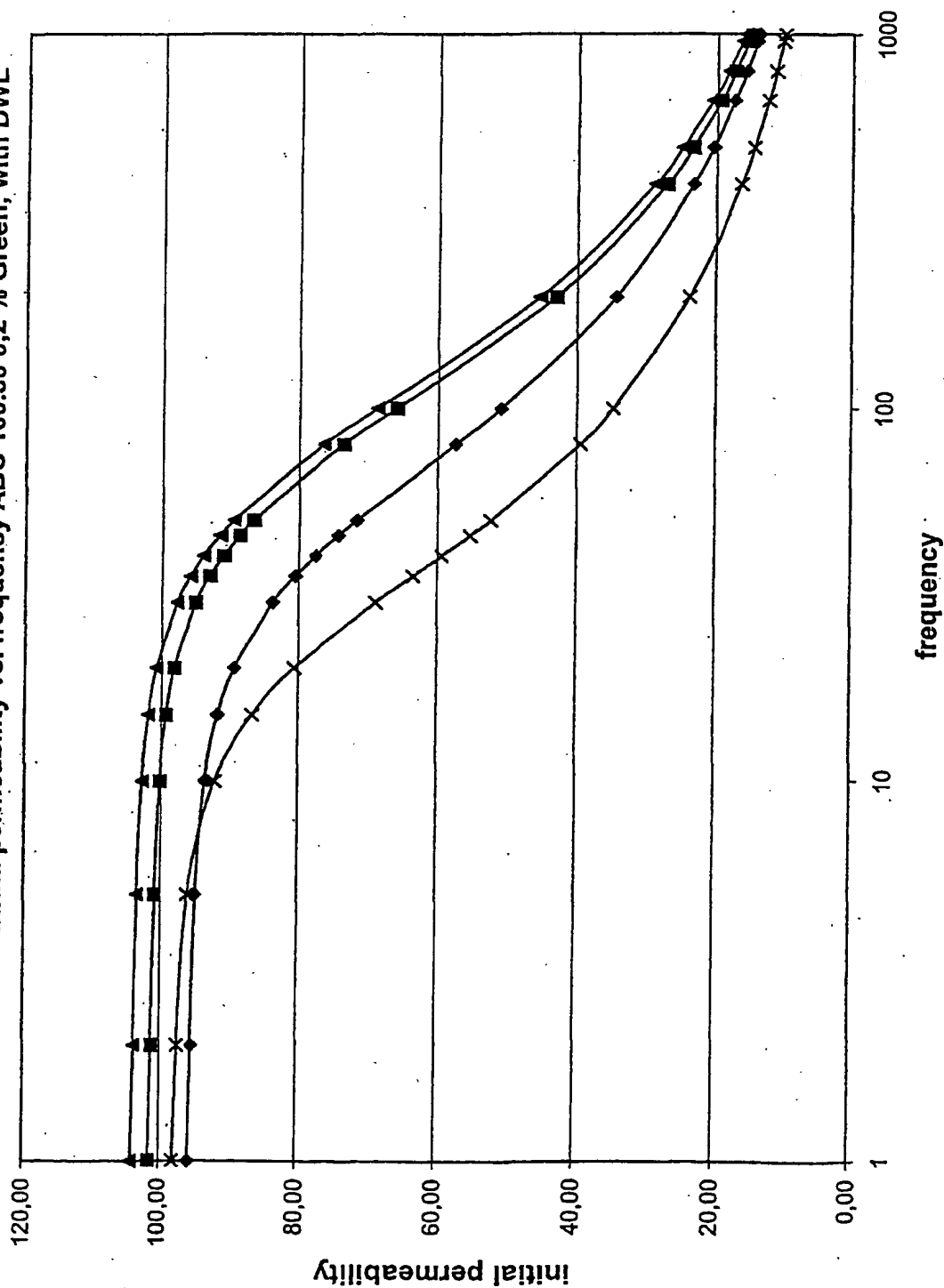
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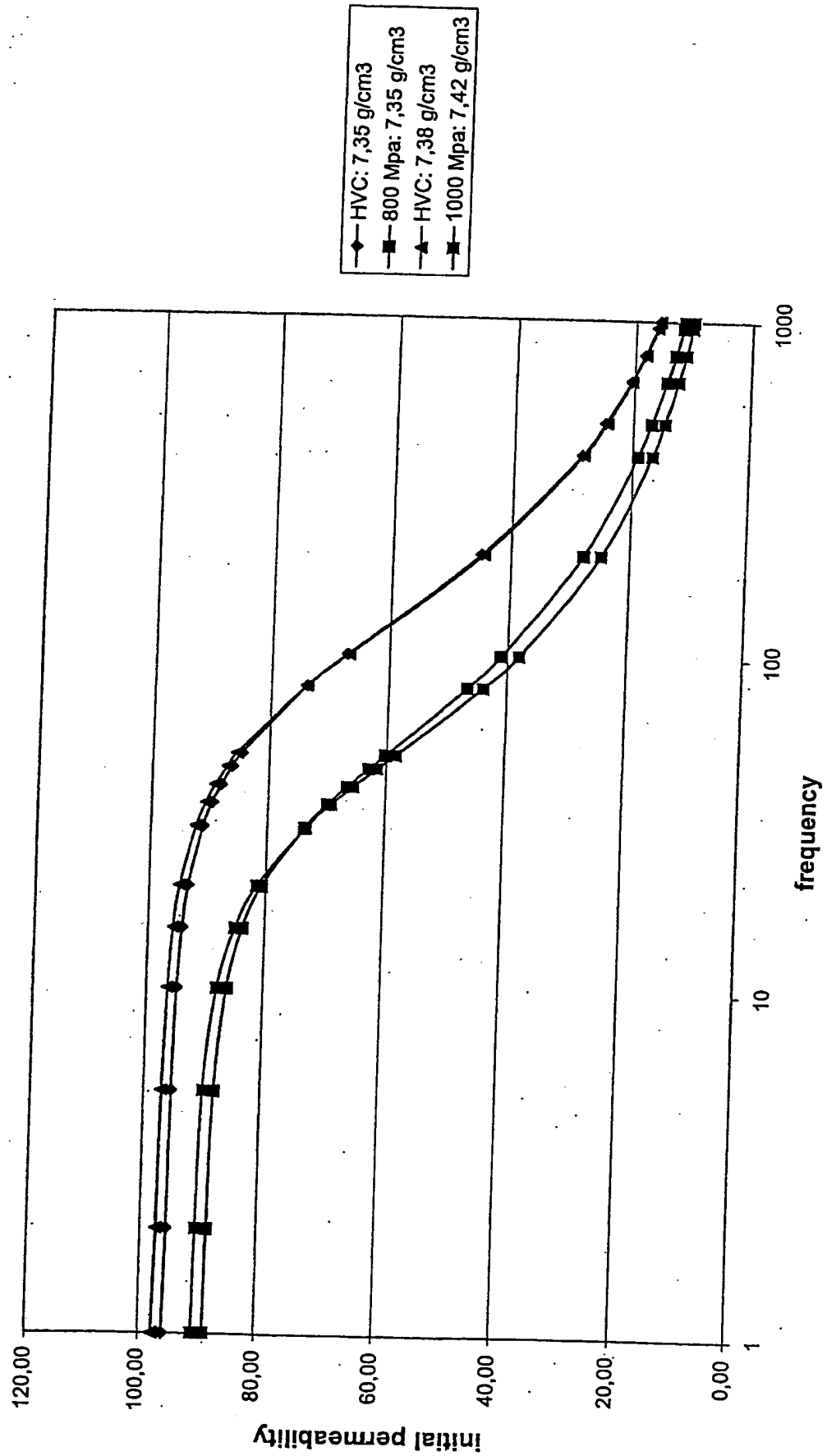
2/3

Initial permeability vs. frequency ABC 100.30 0,2 % Green, with DWL



3/3

Initial permeability vs. frequency ABC 100.30 0,5 % Green, no DWL



INTERNATIONAL SEARCH REPORT

International application No.

PCT/SE 02/01137

A. CLASSIFICATION OF SUBJECT MATTER

IPC7: B22F 3/087, C22C 33/02, H01F 1/24

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC7: B22F, C22C, H01F

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

SE,DK,FI,NO classes as above

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

EPO-INTERNAL

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	WO 9630144 A1 (HÖGANÄS AB), 3 October 1996 (03.10.96) --	1-9
A	EP 0331286 A2 (GENERAL MOTORS CORPORATION), 6 Sept 1989 (06.09.89) --	1-9
A	EP 0331285 A2 (GENERAL MOTORS CORPORATION), 6 Sept 1989 (06.09.89) -- -----	1-9

☐ Further documents are listed in the continuation of Box C.☒ See patent family annex.

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Date of the actual completion of the international search

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INTERNATIONAL SEARCH REPORT
Information on patent family members

06/07/02

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Patent document cited in search report			Publication date	Patent family member(s)		Publication date
WO	9630144	A1	03/10/96	AU	5167096 A	16/10/96
				SE	9501129 D	00/00/00
EP	0331286	A2	06/09/89	JP	1287204 A	17/11/89
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